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Physical sunscreens

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Synopsis

Physical sunscreens have been described as compounds that attenuate radiation through scattering. This work indicates that physical sunscreens can be also strong absorbers of ultraviolet radiation as well as good scatterers of visible light. Compounds like barium sulfate and zinc attenuate radiation at all visible and ultraviolet wavelengths only by scattering. Compounds like titanium dioxide and zinc oxide exhibit a semiconductor optical absorption gap, absorbing most radiation at shorter wavelengths than the gap, while at wavelengths longer than the gap these compounds scatter. Colored iron oxides scatter radiation and simultaneously absorb various visible and ultraviolet wavebands. This knowledge is important in the use of these compounds in sunscreen products because it suggests that while the scattering of a particle can be modified by its environment, the absorption characteristics of a particle cannot be changed.

INTRODUCTION

Sunscreens are classified as chemical or physical depending on whether they absorb specific wavelength bands of radiation or reflect and scatter. When chemical sunscreens are applied to the skin, they usually do not modify the appearance of the skin. When physical sunscreens are applied to the skin, they can be seen on the skin's surface because they reflect and scatter light (1,3). Because physical sunscreens are not wavelength selective, they are recognized as effective ingredients in sunscreen products contributing broad spectral protection.

Greater emphasis is being put on use of physical sunscreens because of regulatory and safety concerns. In Japan, the amount of chemical sunscreen agents that can be used in a product is limited by law. If sunscreen products are to be highly effective, an adequate amount of physical sunscreen must also be employed. At the present time there is no limitation on the amount of physical sunscreen that can be used in a product. Presumably there is no risk to the user from physical sunscreens.

In the United States the OTC drug review examined the safety and efficacy of each

sunscreen agent separately, setting maximum allowable concentrations in products (1). The maximum allowable concentration was determined on the basis of the concentrations that had been used in marketed formulations and that were supported by safety testing for those concentrations. The current policy allows use of combinations of approved agents up to the maximum concentration limits for each agent.

At the time of the OTC review only titanium dioxide was submitted as a physical sunscreen agent for use in sunscreen products. Titanium dioxide, therefore, appears to be the only acceptable physical sunscreen for use in sunscreen products in the United States (1). In the past several years, the availability of physical sunscreens and their quality has increased. The range of particle sizes, especially smaller sizes, has been extended dramatically. Today there are many additional choices of possible physical sunscreen agents. Moreover, we now know better how to design acceptable products incorporating physical sunscreens.

According to Pachak (3), "Physical sunscreens are usually opaque formulations and contain ingredients that are particulate in nature and do not selectively absorb UVR, but when applied as a film, they primarily reflect and scatter UVR and visible radiation because of the size of the particles and thickness of the film." Also in 1986, Kollias *et al.* (2), while establishing the optical specifications for a new spectrophotometer, examined the reflectance characteristics of titanium dioxide, showing that titanium dioxide exhibited a strong absorption edge characteristic of semiconductors at wavelengths just shorter than 400 nm. This observation provided the inspiration for this examination of the definition of physical sunscreens.

METHODS

A Varian Cary 2300 UV/vis spectrophotometer with a Harrick Praying Mantis attachment was used for obtaining diffuse reflectance spectra of physical sunscreen powders. Barium sulfate was used throughout as the reflectance reference (Aldrich Chemical Company #24,355-3). All spectra were measured relative to the BaSO₄ spectrum. Spectra were measured from 250 to 720 nm and stored on magnetic media using a Varian DS-15 data station.

A diffuse reflectance spectrophotometer was also used to obtain similar data from optically thick powder samples (2). This instrument was modified for UV spectrophotometric measurements as follows. A 1000-watt xenon arc (Oriel #6140) was coupled to a Jobin Yvon UV-HL monochromator with the slits set at 1.0 mm (band width of 2 nm), which provided input to a slit-shaped bifurcated silica/silica-clad fiber optic bundle. The common end of the bifurcated bundle had randomly mixed fibers. The other end of the bundle terminated in a slit-shaped fiber array that provided input to a Jobin Yvon H10 monochromator whose output was measured with an Oriel #7070 photomultiplier power supply amplifier. Shutters, stepper motors, and order sorting filter were controlled by and data acquired with a Hewlett Packard Model 150 U microcomputer and a data acquisition controller (HP 3497).

In vivo tests were performed to determine if titanium dioxide and zinc oxide in a clinical product (Rose Cream) provided the protection predicted by the *in vitro* study of sunscreen products. A 10-cm X 10-cm square was drawn on five volunteers' backs. A site within this area was selected and scanned with the fiber optic spectrophotometer prior

to product application. 100 mg of the test product was applied (1 mg/cm^2) and the site was rescanned.

The physical sunscreens examined were:

- Titanium dioxide, Teikoku Kato #3296
- Titanium dioxide, Degussa #P25
- Titanium dioxide, cosmetic grade #8740-40
- Titanium dioxide, Guardian Chemical, ultrafine grade
- Talc, USP
- Talc, USP, micronized (8190-40/19888)
- Zinc oxide USP
- Iron oxide, yellow cosmetic grade
- Iron oxide, black cosmetic grade
- Iron oxide, red cosmetic grade
- Rosa Cream (7.5% zinc oxide + 7.5% titanium dioxide prepared by the Al-Sabah Hospital Pharmacy)

RESULTS

The representative reflectance spectra for titanium dioxide, zinc oxide, talc, and the iron oxides obtained using the Cary 2300 are shown in Figure 1. All spectra are relative to the BaSO_4 reference. Throughout the visible spectrum, all agents tested except the iron oxide reflect 90 to 100 percent of the light relative to barium sulfate. Talc reflects visible and UV similarly to the barium sulfate reference. At wavelengths shorter than 400 nm, titanium dioxide and zinc oxide behave differently from the barium sulfate reference. Both zinc oxide and titanium dioxide reflect less radiation in the ultraviolet than does barium sulfate. The results for the pigmented iron oxides show that these compounds reflect, scatter, and absorb visible wavelengths and behave similarly in the UV radiation.

The reflectance of titanium and zinc oxide powders using the fiber optic remittance spectrometer is shown in Figure 2. The energy gap is shown to be at approximately the same wavelength for both sunscreens.

A representative *in vivo* spectrophotometric test of 1 mg/cm^2 Rosa Cream containing 7.5% zinc oxide and 7.5% titanium dioxide is shown in Figure 3. Note the sharp decrease in reflectance just short of the visible wavelengths. The absorbance of the product is measured by radiation passing twice through the applied product and, therefore, two times larger.

DISCUSSION

The results indicate that there are at least two different types of physical sunscreens: those which only scatter radiation and those that also absorb selected wavelengths. Agents that only scatter radiation are represented by barium sulfate (BaSO_4) and talc. Titanium dioxide (TiO_2), zinc oxide (ZnO), and the colored iron oxides comprise the second group, namely those that scatter some wavelengths and absorb other selected wavelengths of light. This latter group of compounds is potentially very large in number. Both talc and barium sulfate must be visible on the skin's surface if they are to

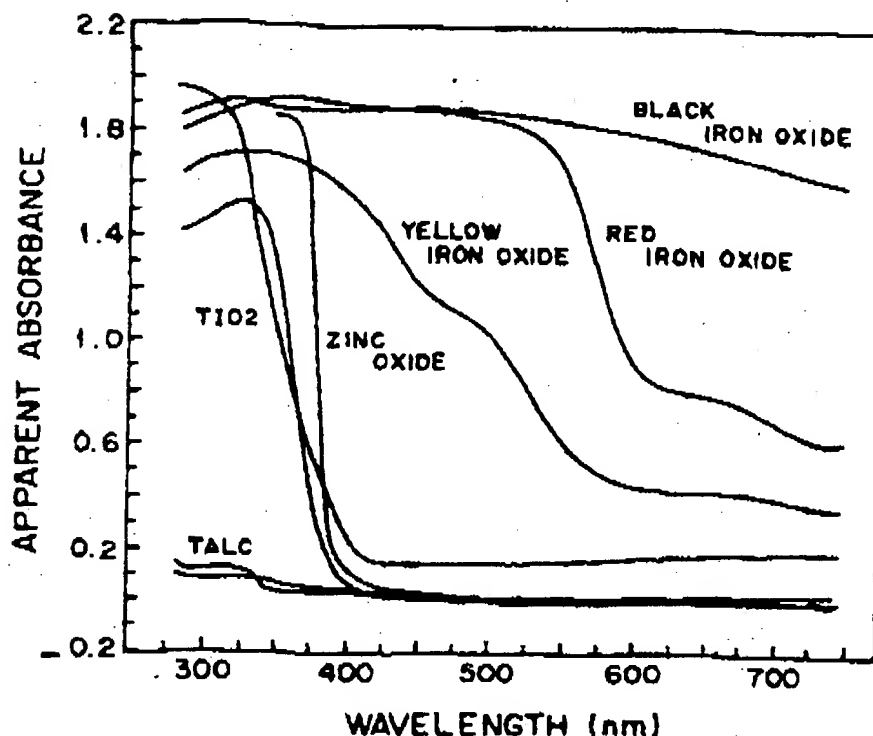


Figure 1. Reflection spectra of physical sunscreen powders. The data plotted shows absorbance vs wavelength. An absorbance of 0.0 corresponds to 100% reflection of the light relative to BaSO_4 ; no absorbance of 1.0 corresponds to 10%; 2.0 corresponds to 1% reflection.

be effective. If they do not appear visible as powders or as a film on the skin, they are not scattering light. It is tempting to speculate that a cosmetically acceptable product providing UV protection could be formulated by matching the index of refraction of barium sulfate in the visible with the index of refraction of the vehicle and having a large difference in index of refraction in the ultraviolet. However, this would be difficult to accomplish, for when the indices of refraction between the scattering particles and the surrounding media are matched in the visible, the formula will disappear on the skin. At this point these particles no longer protect as a sunscreen because they are no longer scattering light. This means that the use of compounds like BaSO_4 and talc as physical sunscreens is limited because the scattering function is susceptible to alteration by the agent's environment.

On the other hand, the use of titanium dioxide or zinc oxide in suncreening products makes a great deal of sense. Both compounds exhibit a very strong absorption band at wavelengths just short of the visible spectrum. This corresponds to the optical band gap of these semiconductor-like materials. At wavelengths shorter than the optical gap, the radiation will excite electrons from the valence band to the conduction band. At wavelengths longer than the optical gap, this mechanism for absorption and dissipation of radiant energy is not available. As the purity is compromised, the absorption edge becomes gradual and the band cut-off is less steep. This is seen in Figure 1, by comparing titanium dioxide curves 1 and 2.

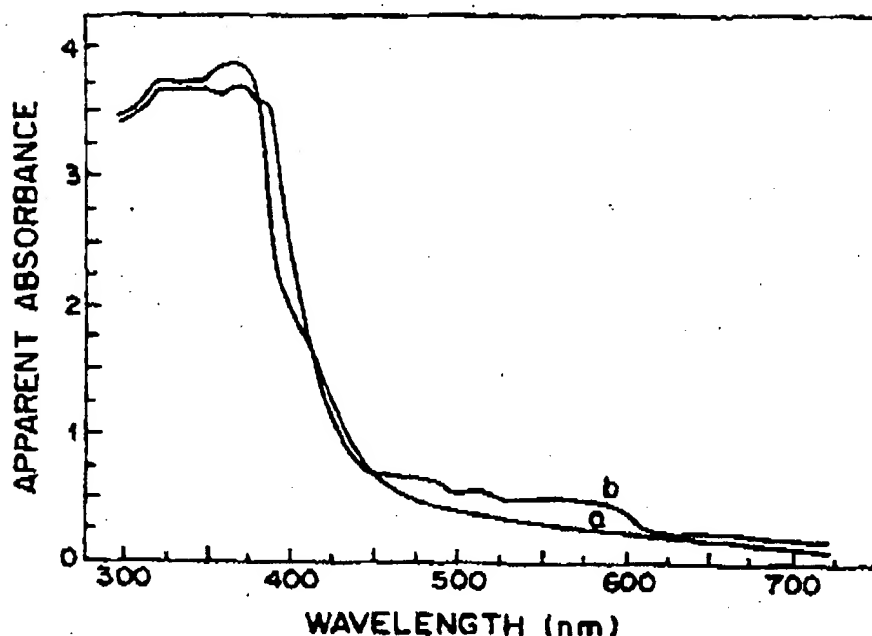


Figure 2. Reflectance spectra of selected sunscreen powders using the fiber optic remittance spectrometer compared to BaSO_4 standard. a) Zinc oxide powder. b) Titanium dioxide powder.

Our *in vivo* data for the ointment applied to skin shown in Figure 3 agree with the *in vitro* work with the ointment and with powders in Figure 1. The very sharp optical gap that begins just below 400 nm can be seen. At wavelengths shorter than the gap, the product should function as a very good broad-spectrum sunscreen. At wavelengths longer than 400 nm, protection will be provided by scattering. The *in vitro* results with powders suggest that high absorbance may be achieved on skin if properly formulated. The benefit of including the absorption of a physical sunscreen that has semiconductor-like properties is that the protection provided by the product will be unchanged by the surrounding media. Formulation techniques can be used to create aesthetically acceptable products using these optical properties.

In sunscreen products where scattering is used as a means of attenuating radiation, the wavelength of the radiation will generally be shorter than the particle diameters of the material employed. This will result in the common Mie type of scattering, which varies relatively slowly with the wavelength of light. If particles are smaller than 0.03 microns in diameter, then Rayleigh scattering will take place.

If scattering is to be used in sunscreen products to attenuate exposure, then the smaller-diameter particles will increase the concentration of particles on the skin's surface and attenuate more radiation for the same concentration of physical sunscreen. The particle radius cannot be indefinitely decreased, however. When the radius becomes substantially smaller than one micron, the scattered light will be preferentially in the forward direction, thereby decreasing the effectiveness of the material.

Iron oxides are intended to provide color to the skin. As can be seen in Figure 1, the

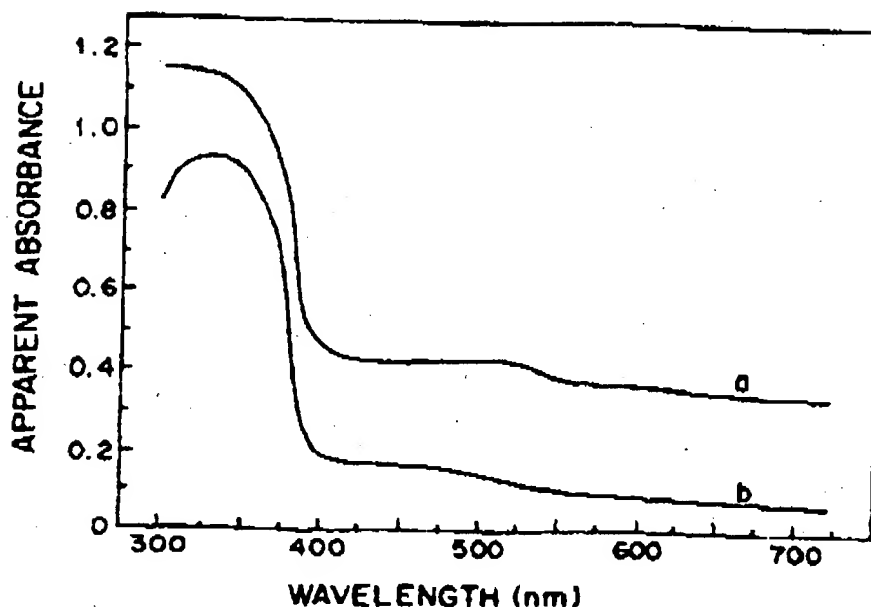


Figure 3. Rosa Cream contains 7.5% TiO_2 and 7.5% ZnO . a) Optically infinitely thick sample with quartz coverslip using remittance spectrometer. b) One mg/cm^2 on human skin using remittance spectrometer. Absorbance is twice as high as expected because radiation must pass twice through the sample before being analyzed.

color is easily detected by measuring reflectance. The low reflectance extends through the ultraviolet. These compounds also should function as physical sunscreens because they absorb the potentially harmful wavelengths.

CONCLUSIONS

Users of physical sunscreen agents should recognize their common properties as particulate powders insoluble in the formulation. In terms of their optical properties they 1) scatter visible and ultraviolet radiation equally well, or 2) scatter visible and absorb ultraviolet, or 3) scatter and absorb visible and ultraviolet to different extents. The scattering properties of these powders can be modified by matching their index of refraction with that of their vehicles, but the absorption properties are characteristic of the bulk material and cannot be modified.

Reflection and scattering of light is effective as a means of protection only if the refractive index of the medium used to disperse the physical sunscreen and that of the physical sunscreen itself are different. The closer these indices are to one another, the lower the screening efficiency. Talc and barium sulfate, which are effective as sunscreen agents only by this mechanism, suffer from this problem.

Iron oxides exhibit electronic absorption bands in the visible region, giving rise to their perceived color. These bands extend into the ultraviolet region, thereby resulting in a potential use as sunscreen agents. Because of the nature and degree of absorption in the

ultraviolet regions, these materials are not widely recognized for their sunscreen potential, despite the fact that they should not suffer from the efficacy problems noted above for agents that only scatter and reflect such light.

Titanium dioxide and zinc oxide each exhibit a strong semiconductor absorption in the ultraviolet. They also scatter and reflect only visible and ultraviolet light. Through careful formulation, aesthetically acceptable products can be made that minimize the scattering and reflection while simultaneously absorbing a significant amount of ultraviolet light because of the clear optical gap. The *in vivo* data presented herein confirm this point. The optical band gap exhibited in the formulated product resulting from the presence of titanium dioxide and zinc oxide can also be observed when product has been applied to the skin.

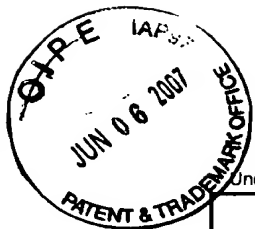
In conclusion, users of physical sunscreen agents should recognize that there are important differences among these agents based upon their mechanism of solar light attenuation. The specific mechanism by which each agent attenuates radiation plays a crucial role in its applicability to particular formulation types. An understanding of the different mechanisms of solar attenuation can potentially yield improved sunscreen efficacy through the combined use of physical and chemical sunscreen agents.

ACKNOWLEDGMENTS

We appreciate the use of the Varian Cary 2300 and Harrick Praying Mantis provided by Dr. M. Shah Jahan, Physics Department, Memphis State University, Memphis, TN 38152.

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- (3) M. A. Pathak, Sunscreens: Topical and systematic approaches for the prevention of acute and chronic sun-induced skin reactions, *Dermatol. Clin.*, 4, 321-33334, (1986).



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Request For Continued Examination (RCE) Transmittal Address to: MS RCE Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450	Application Number	10/982,796
	Filing Date	November 8, 2004
	First Named Inventor	CHAE, Kyung Su
	Art Unit	2871
	Examiner Name	KIM, Richard J.
	Attorney Docket No.	8734.051 C1

This is a Request for Continued Examination (RCE) under 37 CFR 1.114 of the above-identified application.
Request for Continued Examination (RCE) practice under 37 CFR 1.114 does not apply to any utility or plant application filed prior to June 8, 1995, or to any design application.

1. **Submission required under 37 CFR 1.114** Note: If the RCE is proper, any previously filed unentered amendments and amendments enclosed with the RCE will be entered in the order in which they were filed unless applicant instructs otherwise. If applicant does not wish to have any previously filed unentered amendment(s) entered, applicant must request non-entry of such amendment(s).

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2. **Miscellaneous**

a. ☐ Suspension of action on the above-identified application is requested under 37 CFR 1.103(c) for a period of _____ months. (Period of suspension shall not exceed 3 months; Fee under 37 CFR 1.17(i) required)

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SIGNATURE OF APPLICANT, ATTORNEY, OR AGENT REQUIRED			
Name (Print/Type)	Eric J. Nuss	Registration No. (Attorney/Agent)	40,106
Signature		Date	6 June 2007

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Request For Continued Examination (RCE) Transmittal Address to: MS RCE Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450	Application Number	10/982,796
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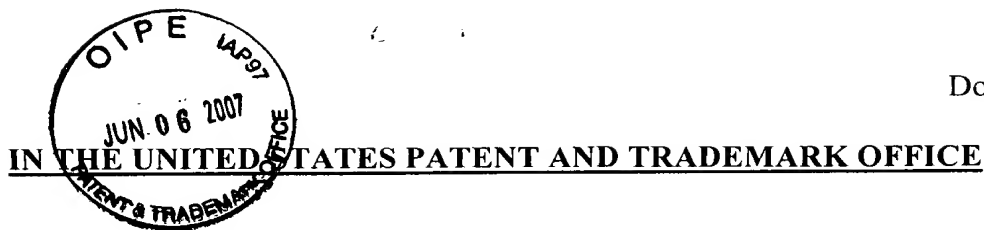
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Name (Print/Type)	Eric J. Nuss	Registration No. (Attorney/Agent)	40,106
Signature		Date	6 June 2007



IN RE APPLICATION: CHAE, Kyung Su

GROUP ART UNIT: 2871

SERIAL NUMBER: 10/982,796

EXAMINER: KIM, Richard J.

FILED: November 8, 2004

**FOR: INDICATOR FOR DECIDING GRINDING AMOUNT OF LIQUID CRYSTAL
DISPLAY PANEL AND METHOD FOR DETECTING GRINDING FAILURE USING
THE SAME**

INFORMATION DISCLOSURE STATEMENT UNDER 37 C.F.R. 1.97

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P.O. Box 1450
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Sir:

Applicant(s) wish to disclose the following information.

REFERENCES

- ☒ The Applicant(s) wish to make of record the references listed on the attached PTO/ SB/08. Copies of the listed references are attached, where required, as are either statements of relevancy or any readily available English translations of pertinent portions of any non-English language references.
- ☐ A check is attached in the amount required under 37 CFR § 1.17(p).

RELATED CASES

- ☐ Attached is a list of applicant's pending applications or issued patents which may be related to the present application. A copy of the patent(s) is attached along with PTO/ SB/08.
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CERTIFICATION

The undersigned certifies that

- ☐ each item of information contained in this information disclosure statement was cited in a communication from a foreign patent office in a counterpart foreign application not more than three months prior to the filing of this statement.
- ☐ no item of information contained in this information disclosure statement was cited in a communication from a foreign patent office in a counterpart foreign application or, to the knowledge of the undersigned, having made reasonable inquiry, was known to any individual designated in 37 CFR § 1.56(c) more than three months prior to the filing of this statement.

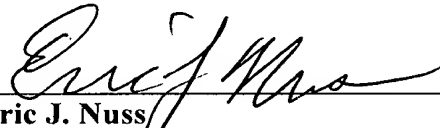
PETITION

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- ☒ Please charge any additional fees for the papers being filed herewith and for which no check is enclosed herewith, or credit any overpayment to deposit account No. 50-0911. A duplicate copy of this sheet is enclosed.

Respectfully submitted,



Eric J. Nuss
Registration No. **40,106**
MCKENNA LONG & ALDRIDGE LLP
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Date: **6 June 2007**



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				Examiner Name	KIM, Richard J.
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				Art Unit	2871
				Examiner Name	KIM, Richard J.
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